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Methyl 12-bromo-13,14-(thiodinitrilo)-deisopropyldehydroabietate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
R factor = 0.040
wR factor = 0.101
Data-to-parameter ratio = 13.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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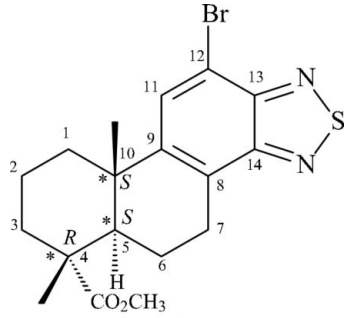
The structure of the title compound, $C_{18}H_{21}BrN_2O_2S$, features a five-membered 2,1,3-thiadiazole ring in which the N atoms are two-coordinate [N—S—N = 101.6 (2)°].

Comment

The reaction of methyl 12-bromo-13,14-dinitrodeisopropyldehydroabietate, the structure of which was reported recently (Pan *et al.*, 2005), with thionyl chloride leads to the formation of methyl 12-bromo-13,14-(2,1,3)-thiadiazolyldehydroabietate, (I) (Fig. 1).



(I)

The bond dimensions of the five-membered ring in (I) compare well with those found in related compounds, such as benzo-2,1,3-thiazole (Luzzati, 1951) and 3,4-diphenyl-1,2,5-thiadiazole (Mellini & Merlino, 1976). The bond dimensions in the other part of the molecule are similar to those of 12-bromo-13,14-dinitrodeisopropyldehydroabietate (Pan *et al.*, 2005) itself.

Experimental

12-Bromo-13,14-diaminodeisopropyldehydroabietate (Fonseca *et al.*, 2004) (1.01 g, 2.65 mmol) was dissolved in dry benzene (15 ml) and the solution was cooled to 273 K. To the solution was added thionyl chloride (0.81 g, 6.84 mmol) dissolved in benzene (15 ml). The mixture was stirred for 0.5 h at 273 K, and then at room temperature for another 1 h before being further refluxed for 12 h. The solvent was removed and the residue was purified by column chromatography on silica gel (light petroleum–ethyl acetate 5:1 *v*/*v*) to give pale-yellow crystals of (I) (0.423 g, 1.034 mmol, 40% yield; mp 370–371 K). Single crystals of (I) were obtained from an absolute acetone solution.

Crystal data

$C_{18}H_{21}BrN_2O_2S$
M_r = 409.34
Monoclinic, $C2$
a = 20.847 (2) Å
b = 12.180 (1) Å
c = 7.1222 (7) Å
β = 101.738 (2)°
V = 1770.5 (3) Å^3
Z = 4
D_x = 1.536 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.45 mm^{-1}
T = 295 (2) K
Block, yellow
0.26 × 0.25 × 0.24 mm

Data collection

Bruker SMART 1000 area-detector diffractometer
φ and ω scans
Absorption correction: none
4679 measured reflections
3068 independent reflections
2244 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.101
S = 0.91
3068 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), with 1368 Friedel pairs
Flack parameter: 0.05 (1)

H atoms were placed in calculated positions, with C—H 0.93–0.98 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C), and were included in the refinement in the riding-model approximation.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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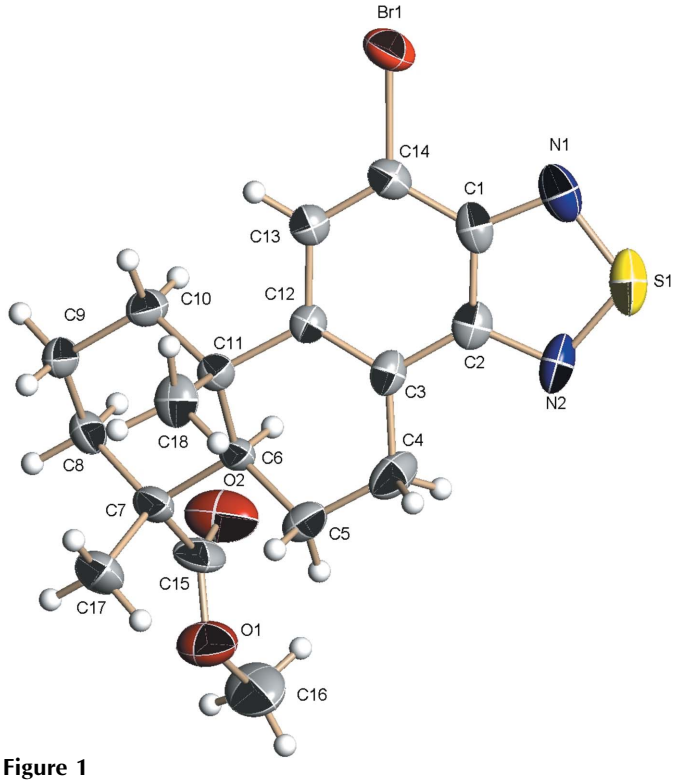

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radii.

References

Bruker (1999). *SAINT* (Version 6.36A) and *SMART* (Version 6.36A). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fonseca, T., Gigante, B., Martiques, M. M., Gilchrist, T. L. & De Clerq, E. (2004). *Bioorg. Med. Chem.* **12**, 103–112.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Luzzati, V. (1951). *Acta Cryst.* **4**, 193–200.
Mellini, M. & Merlino, S. (1976). *Acta Cryst.* B**32**, 1074–1078.
Pan, Y.-M., Zhang, Y., Wang, H.-S., Tong, B.-H., Chen, Z.-F. & Zhang, Y. (2005). *Acta Cryst.* E**61**, o3003–o3005.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.